Detection of Off-Gassing Species in Polymer Composites by Combined TGA/FT-IR

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KEY WORDS

TGA/IR
Polymer Composites
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INTRODUCTION

Specialty polymers and polymer-based composites have found many applications as sealants, gaskets and voltage protection devices in the microelectronics and automotive industries. These manufactured devices must operate at somewhat elevated temperatures and therefore must be free of off-gassing components that may cause product failure. Several of these polymer composites are extremely sensitive to moisture and are known to contain species that are released below or at their decomposition temperatures. Most of these off gases may be moisture, carbon dioxide or byproducts of decomposition and may be present at extremely low levels – less than 0.1% by weight. It is critical to know both, the quantity (available from the TGA and TGA/FT-IR data) as well as identity (available only from the TGA/FT-IR data) of species that are evolved and the temperatures at which these evolutions occur.

EXPERIMENTAL

In this study, we used a thermogravimetric analyzer (TGA) from Seiko Instruments coupled with a Nicolet FT-IR to study a polymer composite and a urethane sealant material. The TGA was used to quantitatively determine the temperature and amount of off gases. The FT-IR was used in the real-time analysis of the chemical composition of these species.

About 33 mg of the polymer molded composite sample was taken in an open platinum pan and heated in the TG from ambient to about 300°C at 20°C/minute. About 40 mg of the urethane sealant material was tested under the same condition. The Seiko TG/DTA, which is a horizontal dual beam design with quartz-lined ceramic furnace, was used in this analysis. A nitrogen purge was set at a flow rate of 50 ml/min. A heated transfer line was attached at the end of the TGA furnace to deliver the evolved gases in to the FTIR analysis chamber. The temperature controller for the transfer line was maintained at a constant temperature of 225°C. The infrared analysis was performed using a Nexus™ FT-IR spectrometer fitted with a TGA interface operated at a constant temperature of 225°C.

RESULTS

Figure 1 shows the TGA and corresponding derivative (DTG) curves for the molded polymer composite plotted as a function of time. Analysis of the DTG curve between 11 and 14 minutes results in a weight loss of 0.03 percent. FT-IR analysis of this extremely small quantity of the off-gases, shown in Figure 2, reveals that it is water. In the second step of the TGA analysis, we measured 0.12 percent weight between 190°C and 280°C, which was determined to be carbon dioxide as shown in the FT-IR scan.

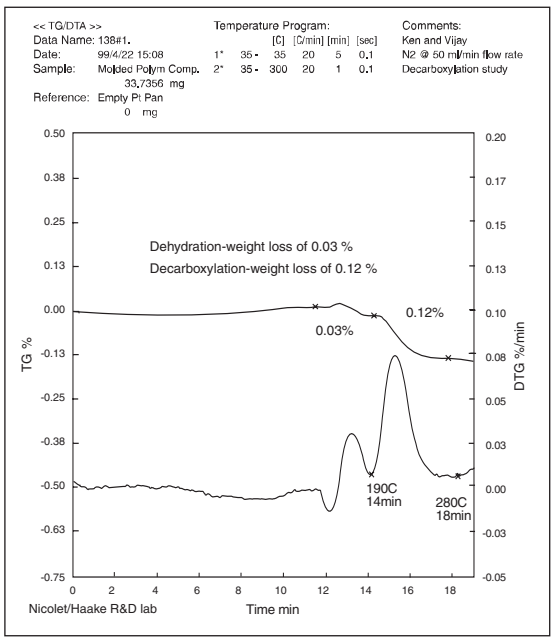

Figure 1: TGA weight loss curve (upper trace) and first derivative of weight loss (lower trace) for molded polymer composite.

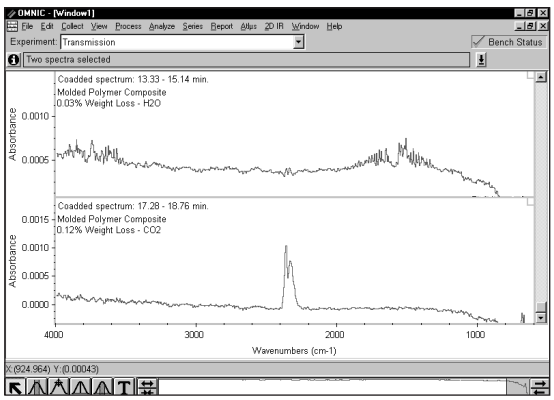

Figure 2: IR spectra for the two weight losses from the molded polymer composite. Upper spectrum is H_20 and lower spectrum reveals CO_2.

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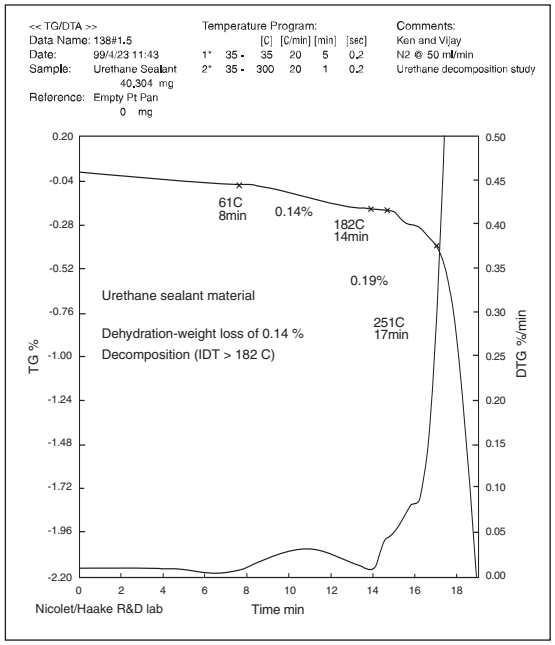

Figure 3: TGA weight loss curve (upper trace) and first derivative of weight loss (lower trace) for urethane sealant material.

Figure 3 is the TG and DTG plots for the urethane sealant material. Analysis of the TGA curve between 8 and 14 minutes shows a weight loss of 0.14 percent. This may be due to the moisture, and FT-IR analysis of the off gases confirms that it is water. Under these experimental conditions, this polymer has an initial decomposition temperature (IDT) of approximately 190°C. FT-IR spectra (shown in Figure 4) of the initial decomposition reveals that it consists of a mixture of methanol, isocyanate and carbon dioxide. Figure 5 shows FT-IR reconstructions of the evolved gases. From these profiles we can see the water loss from off-gassing occurs at lower temperatures and is separate from decomposition of the urethane polymer which begins at temperatures over 190°C.

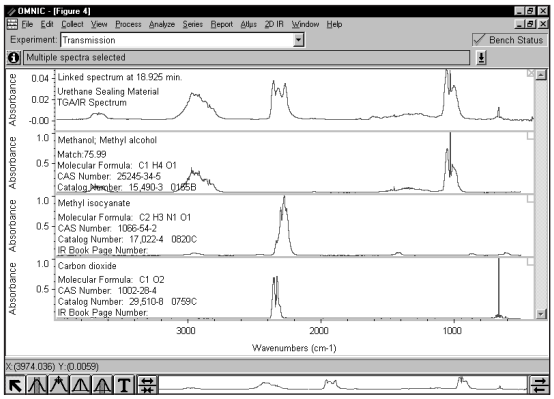

Figure 4: Upper spectrum from TGA/IR evolution at 18.925 minutes. Lower three spectra from Nicolet Vapor Phase Library (methanol, methyl isocyanate and carbon dioxide).

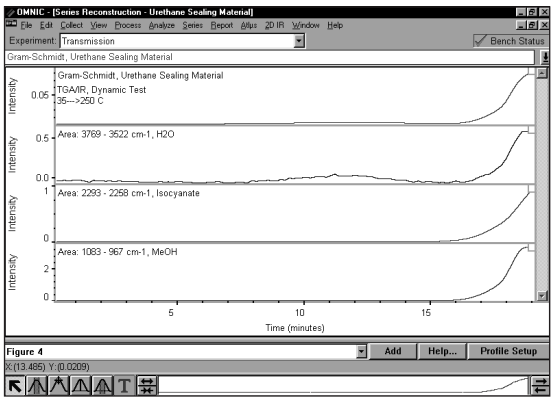

Figure 5: FT-IR reconstructions of the TGA/IR analysis of the urethane sealant material showing GSR (total IR absorbance), H_20, isocyanate and methanol evolution.

CONCLUSION

In the above analysis, we have shown that both TGA and FT-IR are capable of measuring less than 0.10% by weight of off-gassing of polymer materials. In these samples the quartz-lined furnace and highly sensitive and extremely stable baseline of the Seiko TG/DTA were critical for measurement of the amount of weight losses at low levels. The Nicolet Nexus FT-IR with its proprietary folded path TGA flow cell provided the required sensitivity and stability to detect and identify weight losses below 0.10% by weight. The Seiko TG/DTA coupled with the Nicolet Nexus TGA/FT-IR interface provided identification of the off-gassing species and helped differentiate off-gassing and polymer decomposition.

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